Synthesis of Large-Area, Thick, Uniform, Smooth Ultrananocrystalline Diamond Films by Microwave Plasma-Assisted Chemical Vapor Deposition

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Abstract

Applications of Ultrananocrystalline (UNCD) and Nanocrystalline (NC) diamond films have been a common, recent topic of many investigations. The exceptional properties of these films, such as high hardness and chemical inertness combined with their small crystal size and smoothness and excellent mechanical properties such as high Young's modulus, fracture toughness and low coefficient of friction, have suggested application as a protective, hard coating material, a material/substrate for micromechanical systems, a SAW device substrate [1], a robust conducting coating for electrochemical electrodes, and as a freestanding film for vacuum windows or ion beam stripping foils. The commercial success of each of these applications requires the development of repeatable, CVD diamond thin film synthesis processes that are able to deposit these films on a variety of substrates. Additionally, uniform, large area, stress free, thick and thin film deposition is desirable. In this paper we report on the development of process methods and apparatus that enable the uniform and smooth deposition of both thin and thick (> 50 microns) UNCD.

UNCD films are synthesized by microwave plasma-assisted CVD using $Ar/H_2/CH_4$ (i.e. see Zhou et. al. [2]) and $He/H_2/CH_4$ input gas mixtures. The microwave plasma reactor system consists of a variable power, 6KW, and 2.45 GHz power supply, connected to a tunable, cylindrical cavity applicator in which the microwave discharge is confined inside a 12cm diameter cylindrical quartz dome located at the fixed end plate of the applicator [3]. The substrate, typically a three inch diameter silicon wafer, is placed on a stage that is adjusted to place the substrate in direct contact with the microwave discharge. The discharge assumes an intense, hemispherical shape hovering over and in direct contact with the substrate. During each deposition experiment the cavity is tuned and the plasma is adjusted for uniform species and temperature conditions across the substrate. Deposition experiments employ either a thermally floating substrate operation [4] or if desirable the substrate can also be cooled in order to vary the substrate temperature between 550- 850 C. A wide range of experimental conditions, i.e. pressures from 50-250Torr, input powers between 600-2,000 W, variable gas mixtures and flow rates, substrate placement, etc., were explored to identify the optimal conditions that enable the repeatable deposition of uniform, and smooth UNCD films over three inches.

The experimental behavior will be presented. In particular UNCD synthesis, i.e. growth rate, film uniformity and smoothness, are described versus the variables of input power (600-1500W), pressure (80-220 Torr), and input gas chemistry, i.e. H_2 (1-8%) and CH_4 (1-3%). Our experiments have demonstrated that in order to routinely synthesize high quality, smooth and uniform UNCD films the deposition process must be performed in a high purity environment. A large useful UNCD experimental deposition window (80-220Torr, 600-1500W, H_2 1-3%, CH_4 1-2%) was identified. Using 0-1% H_2, i.e. hydrogen deficient conditions, discharges were sustained in contact with the three inch substrates over a 60-200 Torr pressure regime. High quality film deposition was extended to 240 Torr when H_2 concentrations were increased to 1-3%. At a given pressure uniform deposition was achieved by optimizing substrate holder geometry, substrate position, microwave power, and gas chemistry. Film crystal sizes ranged from 3-30nm and film roughness from 11-50 nm and average film thickness over the substrate, which depends on the deposition time, ranged from a few hundred nanometers to over 50 microns. With an additional, modest post deposition mechanical polishing, a surface roughness of 3nm was obtained. Film uniformities of greater than 96% were achieved over three inch silicon wafers. Growth rate increased with increasing pressure, and hydrogen concentration. At pressures of 220 Torr and with CH_4 of 1% and H_2 of approximately 3% the average linear growth rate over the three inches wafer was approximately 1 micron per hour.

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